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Bonding of Restorative Materials to Dentin With Various Luting Agents

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Clinical Relevance

Luting agents have significant effects when used to bond indirect restorative materials to dentin. In the present study, resin cements performed better than water-based cements, especially self-etch and one of two self-adhesive resin cements.

SUMMARY

The aim was to compare eight types of luting agents when used to bond six indirect, laboratory restorative materials to dentin. Cylinders of the six restorative materials (Esteticor Avenir [gold alloy], Tritan [titanium], NobelRondo [feldspathic porcelain], Finesse All-Ceramic [leucite-glass ceramic], Lava [zirconia], and Sinfony [resin composite]) were ground and air-abraded. Cylinders of feldspathic porcelain and glass ceramic were additionally etched with hydrofluoric acid and were silane-treated. The cylinders were luted to ground human dentin with eight luting agents (DeTrey Zinc [zinc phosphate cement], Fuji I [conventional glass ionomer cement], Fuji Plus [resin-modified glass ionomer cement], Variolink II [conventional etch-and-rinse resin cement], Panavia F2.0 and Multilink [self-etch resin cements], and RelyX Unicem Aplicap and Maxcem [self-adhesive resin cements]). After water storage at 37°C for one week, the shear bond strength of the specimens (n=8/group) was measured, and the fracture mode was stereomicroscopically examined. Bond strength data were analyzed with two-factorial analysis of variance (ANOVA) followed by Newman-Keuls' Multiple Range Test (α=0.05). Both the restorative material and the luting agent had a significant effect on bond strength, and significant interaction was noted between the two variables. Zinc phosphate cement and glass ionomer cements produced the lowest bond strengths, whereas the highest bond strengths were found with the two self-etch and one of the self-adhesive resin cements. Generally, the fracture mode varied markedly with the restorative material. The luting agents had a bigger influence on bond strength between restorative materials and dentin than was seen with the restorative material.

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INTRODUCTION

The number of materials available for restoring teeth has increased enormously over recent decades. A variety of indirect restorations are available on the market today, in addition to gold and porcelain-fused-to-metal restorations. These include various ceramic materials, resin composites, and titanium. Retention of some of these restorative materials is dependent on the development of new types of luting agents. For many years, retention of indirect restorations could be attained only by the use of favorable tooth preparations and by mechanical interlocking of the luting agent in irregularities present on the surface of the restoration and the tooth, respectively.[1,2] The luting agent of choice was zinc phosphate cement, which has shown a successful clinical record.[3-6] In 1976, glass ionomer cements were marketed for use as luting agents. These materials are mechanically stronger and less soluble than zinc phosphate cement; they release fluoride and offer chemical bonding to enamel and dentin.[7] However, clinical studies have failed to show any superiority of glass ionomer cements over zinc phosphate cements.[6,8-10]

Indeed, it was the development of dentin adhesives that revolutionized not only the world of direct restorations, but also that of indirect restorations. Adhesive bonding of restorations via a resin cement allowed new types of materials to be used for tooth-colored and/or tooth-preserving restorations (eg, all-ceramic veneers and crowns, resin composite inlays/onlays, resin-bonded bridges).

Adhesives and resin cements have been improved continuously. The focus of research and development has now shifted toward simplifying the use of these materials to reduce application time and technique sensitivity. Thus, resin cements exist in various forms. Some are used following application of an etch-and-rinse adhesive system (conventional "etch-and-rinse resin cements"). Others are used after a self-etching primer ("self-etch resin cements") is applied; still others require no previous pretreatment of enamel and dentin and thus are termed "self-adhesive resin cements."[11,12]

In summary, numerous types of luting agents with overlapping indications are available on the market, and new luting agents are continually being developed. These materials vary in their range of indications, handling sensitivity, and number of application steps. It is important for the practitioner to find luting agents for the dental office that offer simple handling and reliable bonding of restorations to tooth structure.

The purpose of this present study was to compare the various types of luting agents when used to bond different indirect, laboratory restorative materials to human dentin. The null hypothesis was that all luting agents were equally efficient in bonding various restorative materials to dentin.

METHODS AND MATERIALS

Preparation of Restorative Material Samples

Six indirect, laboratory restorative materials were included in the current study (Table 1). Eight cylindrical samples (diameter=5 mm, height=5 mm) were produced from each restorative material according to the respective manufacturers' instructions; this was done at a dental laboratory (CoDENT Dental Laboratory, Aarhus, Denmark). The cylinders were reused throughout the study.

One end of each cylinder was wet-ground by hand on silicon carbide (SiC) paper #500 (LaboPol-1/SiC paper, diameter 200 mm, Struers A/S, Ballerup, Denmark) and was air-abraded with 50 μm alumina particles for 10 seconds at a distance of 10 cm and a

Table 1: *Restorative Materials Included in the Study*

Restorative Material	Name and Specifications	Manufacturer
Gold alloy	Esteticor Avenir (Au 84% weight, Pt 10.9% weight, Pd 2.4% weight, Ag 0.2% weight)	Cendres & Métaux, Biel-Bienne, Switzerland
Titanium	Tritan Pure Titanium grade 1, ISO 5832-2 (Ti ≥ 99.5% weight, Fe, O, H, N, C)	Dentaurum, Ispringen, Germany
Feldspathic porcelain	NobelRondo Feldspathic crown and bridge porcelain	Nobel Biocare, Göteborg, Sweden
Glass ceramic	Finesse All-Ceramic Leucite-reinforced glass ceramic	Dentsply Ceramco, Burlington, NJ, USA
Zirconia	Lava Yttrium stabilized zirconium-oxide ceramic core material	3M ESPE, Seefeld, Germany
Resin composite	Sinfony Indirect microhybrid laboratory composite	3M ESPE, Seefeld, Germany

pressure of 4.2 bar (Basic Duo, Renfert, Hilzingen, Germany). Air abrasion was followed by rinsing with 96% ethanol and air-drying. Cylinders of feldspathic porcelain and glass ceramic were then etched with hydrofluoric acid (Top Dent Porcelain Etch Gel 9.6%, DAB Dental, Upplands Väsby, Sweden) for 2 minutes, rinsed with water for 2 minutes, and air-dried. Etching was followed by application of a silane coupling agent (Top Dent Bond Enhancer Silane, DAB Dental), which was left to dry for 4 minutes. Before reuse, cylinders were resurfaced according to the previous procedures.

Preparation of Dentin Surfaces

Human molars that had been kept in 0.5% chloramine aqueous solution since the time of extraction were cleaned of debris and embedded in self-curing epoxy resin (Epofix, Struers). After the epoxy resin had set, the mesial surface of each tooth was wet-ground using sequential SiC papers from #220 to #500 (Struers) to remove enamel and provide a flat dentin surface.

Luting Procedures

The cylinders of each indirect restorative material were luted to the ground dentin surfaces with one of eight luting agents (Table 2), according to the luting procedures listed in Table 3. The luting agent (~0.05 g) was applied to the prepared end of the cylinder, which then was brought into contact with the dentin surface. The cylinder was pressed against the dentin surface using a brass holder and a brass rod with a force of 2 N. The light-curing luting agents were given a tack cure (Bluephase LED light-curing unit [high power mode], Ivoclar Vivadent AG, Schaan, Liechtenstein) of 1 second at each of two opposite sides of the cylinder. Following removal of excess luting agent, the luting agent was light-cured for a total of 40 seconds, 10 seconds at each of four sites around the cylinder (Bluephase LED light-curing unit [high power mode], Ivoclar Vivadent AG). For non–light-curing luting agents, the excess was removed immediately after the cylinder was seated. All luted specimens were left at room temperature for 10 minutes after the start of mixing of the luting agent. The specimens then were removed from the holder and were transferred to a water bath at 37°C. The specimens in the 48 groups were produced in random order, and light power intensity of the curing unit was controlled to be $\geq$950 mW/cm^2 before the luting procedures were performed (Demetron LED Radiometer, SDS Kerr, Middleton, WI, USA).

Table 2: *Luting Agents Included in the Study*

Luting Agent	Name and Specifications	Manufacturer
Zinc phosphate cement	DeTrey Zinc Fine grain zinc phosphate cement (powder: zinc oxide 88% wt, magnesium oxide 9% wt; liquid: phosphoric acid 0.59% wt)	Dentsply DeTrey, Konstanz, Germany lot no.: powder 0602002203; liquid 0604000692
Conventional glass ionomer cement	Fuji I Capsule Self-cure, radiopaque glass ionomer cement	GC Corporation, Tokyo, Japan lot no.: 0607221
Resin-modified glass ionomer cement	Fuji Plus Capsule Self-cure, radiopaque resin-modified glass ionomer cement	GC Corporation, Tokyo, Japan lot no.: 0606161
Conventional etch-and-rinse resin cement	Variolink II ▪ Total Etch, Excite DSC ▪ Paste/Paste (Base/Catalyst) adhesive resin cement	Ivoclar Vivadent, Schaan, Liechtenstein lot no.: Total Etch H30565; Excite DSC H02813; Base J08466, Catalyst J08416
Self-etch resin cement	Panavia F2.0 ▪ ED Primer II (Liquid A/Liquid B) ▪ Paste/Paste (Paste A/Paste B) dual-curing adhesive resin cement	Kuraray Medical, Okayama, Japan lot no.: ED Primer II A 00229A, ED Primer II B 00107A; Paste A 00213A, Paste B 00117A
Self-etch resin cement	Multilink ▪ Multilink Primer (Primer A/Primer B) ▪ Automix (Base/Catalyst) dual-curing adhesive resin cement	Ivoclar Vivadent, Schaan, Liechtenstein lot no.: Primer A J13665, Primer B J16444; Cement J16459
Self-adhesive resin cement	RelyX Unicem Aplicap ▪ Powder/Liquid dual-curing self-adhesive resin cement	3M ESPE, Seefeld, Germany lot no.: 261642
Self-adhesive resin cement	Maxcem ▪ Automix (Base/Catalyst) dual-curing self-adhesive resin cement	Kerr, Orange, CA, USA lot no.: 453579

Table 3: *Luting Procedures Included in the Study*

Luting Agent	Treatment Steps (According to Manufacturers' Instructions)	Time
DeTrey Zinc	Portions of powder added one by one to liquid, spatulating DeTrey Zinc application	90 s
Fuji I	Capsule activation Capsule mixing (Dentsply DeTrey) Fuji I application	10 s
Fuji Plus	Capsule activation Capsule mixing (Dentsply DeTrey) Fuji Plus application	10 s
Variolink II	Total Etch gel (37% phosphoric acid) Water spray Excite DSC application Mixing Variolink II Base and Catalyst 1:1 Variolink II application	10–15 s >5 s + air-dry 10 s + air-dry 10 s
Panavia F2.0	Mixing ED Primer II Liquid A and B 1:1 ED Primer application Mixing Panavia F2.0 Paste A and B 1:1 Panavia F2.0 application	30 s + air-dry 20 s
Multilink	Mixing Multilink Primer A and B 1:1 Multilink Primer application Multilink application through automix tip	15 s + air-dry
RelyX Unicem	Capsule activation Capsule mixing (Dentsply DeTrey) RelyX Unicem application	>2 s 15 s
Maxcem	Maxcem application through automix tip	

Shear Bond Strength and Fracture Mode

After 1 week of water storage, the specimens were subjected to a shear bond strength test using a crosshead speed of 1 mm/min in a universal testing machine (Instron 5566, Instron Ltd, High Wycombe, UK). After the shear bond strength test had been done, the fracture mode was determined using a stereomicroscope at 18× magnification (Ernst Leitz No. 509088, Ernst Leitz GmbH, Wetzlar, Germany).

Statistical Analysis

Shear bond strength data were statistically analyzed with a two-factorial analysis of variance (ANOVA) (independent variables: restorative material and luting agent; dependent variable: shear bond strength) and Newman-Keuls' Multiple Range Test, with $\alpha=0.05$ as the level of significance. The fracture mode was analyzed descriptively.

RESULTS

The results of the bond strength tests are depicted in Table 4. Statistical analysis showed a significant effect of restorative material ($p=0.0006$) and of luting agent ($p<0.0001$), as well as a significant interaction between the two variables ($p<0.0001$). The interaction implies that no restorative material gave consistently the highest or the lowest bond strength, and that no luting agent was universally superior, irrespective of restorative material. Thus, the luting agents that gave the highest bond strengths varied, depending on the restorative material, and were as follows: 1) gold alloy: RelyX Unicem, Panavia F2.0, and Multilink; 2) titanium: Variolink II, RelyX Unicem, Panavia F2.0, and Multilink; 3) feldspathic porcelain: RelyX Unicem, Panavia F2.0, and Multilink; 4) glass ceramic: RelyX Unicem, Panavia F2.0, and Multilink; 5) zirconia: RelyX Unicem and Panavia F2.0; and 6) resin composite: Multilink. Based on their general performance, the luting agents can be divided into three groups; those in the first group gave the lowest bond strengths, and those in the third group gave the highest bond strengths—group 1: DeTrey Zinc, Fuji I, Maxcem, and Fuji Plus; group 2: Variolink II; and group 3: RelyX Unicem, Panavia F2.0, and Multilink.

Two types of fracture mode were observed: adhesive fracture between restorative material and luting agent, and adhesive fracture between luting agent and dentin. The results of fracture mode determination are shown in Table 5 as the percentage of adhesive fractures between luting agent and dentin. For most luting agents, the percentage varied markedly depending on the restorative material; however, the percentage was rather stable for a few luting agents: Maxcem consistently debonded from the dentin, whereas Multilink more often than not debonded from the restorative material, with only a few cases of adhesive fracture between the luting agent and the dentin.

Table 4: *Strength of Bond (MPa) of Restorative Materials to Dentin Mediated by Various Luting Agents (n=8)*[a,b]

Restorative material	Luting Agent							
	DeTrey Zinc	Fuji I	Fuji Plus	Variolink II	Panavia F2.0	Multilink	RelyX Unicem	Maxcem
Gold alloy	1.4 (0.4)[AB]	3.1 (1.2)[ABCD]	4.7 (2.5)[BCD]	9.8 (2.6)[FGH]	13.2 (2.2)[HIJKL]	13.9 (2.7)[JKL]	10.9 (2.8)[GHIJ]	4.2 (1.3)[BCD]
Titanium	1.8 (0.7)[ABC]	3.5 (1.7)[ABCD]	6.7 (2.7)[DEF]	11.5 (2.1)[GHIJ]	13.8 (4.1)[JKL]	11.6 (2.7)[GHIJ]	11.4 (1.5)[GHIJ]	5.6 (2.1)[CD]
Feldspathic porcelain	0.8 (0.4)[AB]	1.2 (0.4)[AB]	3.4 (1.9)[ABCD]	4.0 (2.9)[BCD]	10.3 (1.8)[GHIJ]	11.0 (1.9)[GHIJ]	11.2 (2.2)[GHIJ]	4.3 (1.2)[BCD]
Glass ceramic	1.3 (0.6)[AB]	1.3 (0.9)[AB]	3.8 (1.5)[ABCD]	8.8 (1.8)[EFG]	10.6 (2.8)[GHIJ]	13.5 (2.9)[IJKL]	9.9 (2.5)[FGHI]	4.0 (1.5)[BCD]
Zirconia	2.2 (0.5)[ABC]	4.6 (2.6)[BCD]	9.2 (3.2)[EFG]	6.5 (1.9)[DE]	15.0 (3.7)[KL]	6.2 (1.3)[DE]	13.2 (3.2)[HIJKL]	4.2 (2.1)[BCD]
Resin composite	0.1 (0.2)[A]	3.4 (1.2)[ABCD]	2.9 (0.9)[ABCD]	9.4 (3.1)[EFG]	11.9 (3.6)[GHIJK]	16.1 (4.3)[L]	9.0 (1.9)[EFG]	4.6 (2.1)[BCD]

[a] *Identical superscript letters indicate no statistically significant differences between groups.*
[b] *Mean values and standard deviations.*

Table 5: *Percentage (%) of Adhesive Fractures Between Luting Agent and Dentin (n=8)*

Luting Agent	DeTrey Zinc	Fuji I	Fuji Plus	Variolink II	Panavia F2.0	Multilink	RelyX Unicem	Maxcem
Restorative material								
Gold alloy	25	0	0	38	13	0	0	100
Titanium	63	25	13	50	100	0	75	100
Feldspathic porcelain	13	0	13	75	13	13	13	100
Glass ceramic	0	13	13	50	75	13	38	100
Zirconia	63	0	50	38	88	0	75	100
Resin composite	0	50	0	50	88	38	88	100

DISCUSSION

The present study investigated the bond strength of six indirect restorative materials luted to dentin with eight luting agents. In general, the zinc phosphate cement showed the lowest bond strength values. This finding is in agreement with findings of previous studies.[13–16] The low bond strength of zinc phosphate cement can be explained by a well-documented lack of adhesion and the absence of a chemical bond to tooth structure.[17,18] As a clinical consequence, zinc phosphate cement can be recommended only in situations of tooth preparations with substantial tooth structure left. This applies to the conventional glass ionomer cement Fuji I as well. Although glass ionomer cements do bond chemically to dentin,[19] the bond strength values of Fuji I were not significantly higher than those of zinc phosphate cement. The resin-modified glass ionomer cement Fuji Plus bonded significantly better than zinc phosphate cement for only two materials: titanium and zirconia. The lowest bond strength values were obtained when Fuji I or Fuji Plus was used for luting feldspathic porcelain or glass ceramic to dentin and, in the case of Fuji Plus, for luting resin composite to dentin.

Regarding the fracture mode, the two glass ionomer luting agents presented a low adhesive capacity for these restorative materials with 87% to 100% adhesive fractures between luting agent and restorative material. The low bonding performance is in accordance with a study reported by Piwowarczyk and others, in which resin-modified glass ionomer cements yielded significantly lower shear bond strength values than resin-based luting agents.[15] However, when bonding zirconia to dentin, Fuji Plus yielded statistically equal bond strength values, as did the conventional etch-and-rinse resin cement Variolink II or the self-etch resin cement Multilink. Nevertheless, the fracture modes differed in that Multilink always failed at the zirconia surface, whereas Fuji Plus (with 50% adhesive fractures between luting agent and zirconia) seemed to bond equally well to zirconia and to dentin. Fuji Plus even yielded significantly higher bond strength values with zirconia than did the self-adhesive resin cement Maxcem. Indeed, the bonding performance of Maxcem generally resembled that of the zinc phosphate and glass ionomer cements and not that of the other resin cements. Furthermore, regardless of the restorative material, Maxcem was the only luting agent to present 100% adhesive fractures between luting agent and dentin, indicating a highly limited self-adhesive capacity to dentin. This poor bonding capacity to dentin of Maxcem has been reported in other studies as well.[12,20,21]

Whereas Variolink II and Multilink generally provided higher bond strengths of restorative materials to dentin, these two resin cements did not perform significantly better than Maxcem when bonding zirconia to dentin. Variolink II also did not perform better than Maxcem when bonding feldspathic porcelain to dentin.

When the etch-and-rinse resin cement Variolink II was compared with the two self-etch resin cements, both Panavia F2.0 and Multilink generally showed higher bond strength values than did Variolink II. Previous studies have also reported higher bond strength of Panavia F2.0 when compared with Variolink II: In one case, the bond strength values of Panavia F2.0 were significantly higher than those of Variolink II with the Syntac adhesive system. This result was due to an exceptionally high number of pretesting failures among Variolink II specimens.[22] In another case, Panavia F2.0 yielded a significantly higher bond strength when compared with Variolink II with the Excite DSC adhesive system to root canal dentin.[23] In a third study, Panavia F2.0 yielded higher bond strength values than Variolink II with the Excite DSC adhesive system; however, no statistically significant difference was noted.[12]

When Multilink is compared with Variolink II, the superiority of the self-etch resin cement Multilink noted in the present study is contradicted by the results of other studies. Toman and others found Multilink to show significantly lower shear bond strength values than Variolink II when used with the Excite DSC adhesive system.[24] Moreover, Zhang and Degrange reported that Variolink II and Multilink promoted bonds of equal magnitude between various restorative materials and dentin.[25] One possible explanation for the discrepancies between studies is that the methods applied were different (eg, whether or not thermocycling was applied).

In terms of the two self-etch resin cements, Mirmohammadi and others indicated either equal or significantly lower bond strength values of Multilink when compared with those of Panavia F2.0, depending on the test method used.[26] In another study, which used Panavia F and not Panavia F2.0, Multilink also yielded lower bond strength.[27]

The performance of the etch-and-rinse cement Variolink II in comparison with the self-adhesive cement RelyX Unicem on dentin is also controversially discussed in the literature. One shear bond strength study reported that Variolink II with Excite DSC performed significantly better than did RelyX Unicem;[28] another study indicated that Variolink II with the Syntac adhesive system showed significantly higher bond strength values than RelyX Unicem.[29] On the other hand, several studies confirm the findings of the present work, in which Variolink II yielded equal or significantly lower bond strength values when compared with RelyX Unicem.[12,22,23,30,31]

Statistically equal bond strengths to dentin have also been reported between self-etch resin cements and the self-adhesive resin cement RelyX Unicem.[12,22,29,30,32] This finding is consistent with the present study in 10 of 12 possible comparisons. The exceptions were that RelyX Unicem performed significantly worse than did Multilink in bonding to resin composite, and that Multilink performed significantly worse than did RelyX Unicem in bonding to zirconia. Thus, RelyX Unicem and Panavia F2.0 yielded equal bond strengths with all restorative materials.

To sum up, the present study showed that the combination of various luting agents and restorative materials had varying effects in the bond strength to dentin, and the null hypothesis was rejected. The present results were obtained following water storage for one week. It may be that thermocycling or long-term water storage would have altered the performance of the luting agents.

CONCLUSIONS

Based on the current results, it can be concluded that:

- The resin cements gave higher bond strengths than did the water-based cements DeTrey Zinc, Fuji I, and Fuji Plus.
- The self-etch resin cements Panavia F2.0 and Multilink generally gave higher bond strengths than did the conventional etch-and-rinse resin cement Variolink II.
- The self-adhesive resin cement RelyX Unicem performed as well as the self-etch resin cements Panavia F2.0 and Multilink, whereas the other self-adhesive resin cement Maxcem performed equally to the water-based cements DeTrey Zinc, Fuji I, and Fuji Plus.

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Conflicts of Interest

The authors declare no conflicts of interest, real or perceived, financial or nonfinancial.

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